RESEARCH AND DEVELOPMENT ON CELLS WITH BELLOWS CONTROLLED ELECTROLYTE LEVELS

BY

AMMEGERIALZBERG

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(CODE)

THIRD PROGRESS REPORT DECEMBER 10, 1964 MAY 10, 1965

PREPARED FOR

GODDARD SPACE FLIGHT CENTER

CONTRACT NAS 5-3813

THE ELECTRIC STORAGE BATTERY COMPANY
MISSILE BATTERY DIVISION
RALEIGH, NORTH CAROLINA

THIRD PROGRESS REPORT

ON

RESEARCH AND DEVELOPMENT
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SUMMARY

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Electrolyte level control has been obtained in sealed 6 AH nickel cadmium cells with both metal and plastic bellows during 24 hour orbit cycling. Electrolyte level has varied from near full flood at end of discharge to 40% immersion at end of charge. An auxiliary electrode and increased amount of charged cadmium active material have improved oxygen recombination.

Polypropylene film, heat sealed into small partially inflated pillows, has shown excellent bellows action in both cell tests and flexing tests. No failures occurred after flexing under 20% KOH for a total of 26,500 cycles: 7700 cycles at room temperature, 7800 cycles at 0°C and 11,000 cycles at 40°C. Selective grouping of pillow units can be used to "tailor" a bellows for sensitivity to action ranging from chiefly compression to chiefly expansion.

Sealed 8 AH silver cadmium cells designed for optimum recombination and bellows action are under construction. The bellows compartment amounts to less than one-fourth of cell height. Charge control by a bellows-switch combination is under design for this cell.

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INTRODUCTION

Work in the third quarter has been concerned with all three developmental tasks. Experience thus gained is applicable to the design of deliverable cells under Task 4.

The test program of <u>Task 1</u>, the Ni-Cd system, is under way. Test cells were designed to enhance oxygen recombination by the use of 20% KOH electrolyte, by a higher ratio of charged negative to charged positive active material and by use of auxiliary electrodes.

Two parameters have demanded further consideration for bellows equipped cells - (1) the electrolyte level at atmospheric pressure before sealing and, (2) the imposed cell pressure and the electrolyte level after seal.

A clamped gasket seal for the test cells has been adopted to allow versatility in changing such parameters as the separator. This seal has permitted the selection of optimum conditions for the final cycle tests. Experience in fabricating plastic pillow units has made it possible to "tailor" a bellows to the type of action needed.

The design of a sealed cell with a practical rectangular bellows configuration has been worked out for the Ag-Cd system of <u>Task 2</u>. Twenty-four of these cells with four different separator systems and a plastic bellows substitute, a series of rectangular pillows, are under construction for the test program. A slotted pillow "cage" serves to confine the buoyant pillows and affords easy passage for liquids and freedom of movement for for the pillows. Thin auxiliary oxygen recombination electrodes, placed

against the outer plates of the cell pack, are connected to a third cell terminal to allow monitoring of the recombination rate.

The cell design of Task 2 is being adapted to the bellows-switch control for <u>Task 3</u>. A combination of metal bellows with mechanical switch is under design study with the Servometer Corporation, the supplier of electroformed nickel bellows. A plastic bellows-pressure switch combination is under study with the Metals and Controls Division of the Texas Instrument Company, supplier of subminiature switches.

2. WORK ACCOMPLISHED DURING THIRD QUARTER

2.1 <u>Task 1 - Feasibility of Electrolyte Level Control by Bellows In A Sealed Cd/KOH/NiOOH Cell.</u> - Parameter studies in the second quarter with 4 AH sintered plate cells established the advantage of using 20% KOH electrolyte and thin open weave separator materials for promoting oxygen recombination in a sealed cell with bellows controlled electrolyte level.

Further optimization was foreseen by increasing the ratio of charged negative active material to charged positive active material (1) and by use of auxiliary oxygen electrodes connected to the negative group (2). All of these factors have been incorporated into the 6 AH cells now engaged in the evaluation program. The evaluation test schedule as described in the Second Quarterly Report (3) is being followed but the selection of parameters to be used for optimizing performance has been delayed. This change in the program plan will broaden the scope of study without undue expenditure of time, effort or materials. Essentially it provides for evaluation of the following:

- a. Four separator systems instead of the original two.
- b. Variations in construction and arrangement of the plastic pillows.
- c. A comparative check on oxygen electrode effectiveness.
- d. Electrolyte level before and after seal.

Bellows study has included calibration of both sizes of the purchased nickel bellows along with the development and calibration of a suitable non-metallic bellows substitute.

2.1.1 Continuation of Parameter Study. -

2.1.1.1 Modification of Cloth Separator By Filaments. -

a. <u>Modification Goal</u> = Improvement in electrolyte drainage without impairment of discharge or overcharge characteristics.

b. Separator Description

Filament = 11 mil black polypropylene monofilament. Three strands wrapped vertically
around the positive plates and evenly spaced.

Cloth = AGB, Type 8M, 96 mesh, nylon monofilament cloth, 4 mils thick, wrapped in double
layer around the positive plate with its strands.

c. <u>Test Cells</u> = Two unsealed 4 AH, sintered plate,

nickel cadmium cells, Cell No. 3 with 25% KOH and Cell No. 4 with 20% KOH.

d. <u>Test To Measure Effect of Filament and Cloth</u> <u>Separators on Discharge Characteristics at</u> Three Different Electrolyte Levels. -

(1) Test Procedure. -

Charge (cells flooded) = at 400 ma for
16 hours (1.6C).

<u>Discharge</u> (at selected electrolyte level) at 2.0 amps (C/2) to 1.00V.

Discharge No. 1 at 100% electrolyte level (flooded)

Discharge No. 2 at 50% electrolyte

level. Discharge to cutoff V, then

flood cell and run residual discharge
to cutoff V.

Discharge No. 3 at 10% electrolyte level.

(2) <u>Test Results</u>. -

Figure 1 is a plot of the discharge voltage characteristics. It shows practically no effect at 50% level but indicates a definite drop in capacity to less than 1/2 flooded capacity at 10% electrolyte level. The curve for 25% KOH suggests a somewhat greater tendency toward level sensitivity.

(3) Discussion. -

When compared with the data obtained for net separators and for nylon cloth without filament, shown in Figure 5 of the Second Progress Report, (3) test results show that filaments can be used without impairment of discharge characteristics. It seems likely also, that various degrees of level effect could be obtained by use of different filament diameters.

e. <u>Test to Measure Effect of Filament and Cloth Separators</u> <u>On Overcharge Characteristics of Sealed Cells With Plastic</u> Pillow Level Control. -

(1) Sealed Test Cell Characteristics. -

Bellows = 4 mil polypropylene film heat sealed into rectangular pillow, 2 x 1-7/8", with 1.5 cc trapped air.

Electrolyte Quantity = 10% level at atmosphere
 pressure.

Seal Conditions = Cells evacuated to 28" Hg at seal.

Resulting Electrolyte Level:

Cell No. 3 = 84% immersion

Cell No. 4 = 86% immersion

(2) Test Procedure. -

<u>Charge</u>: at 200 mA (C/20) and continue into overcharge to equilibrium.

Monitor: Cell voltage, pressure and electrolyte
level.

(3) <u>Test Results</u>. -

Figure 2 shows the variation of voltage, pressure and level over a 58 hour period. With onset of overcharge the electrolyte level in both cells dropped below 10%, and by 30 hours the level had stabilized at about 1%.

- Cell No. 3, with 25% KOH, showed steadily increasing voltage and pressure, reaching 1.6 V and 40 PSIA by 44 hours and 1.62 V and 52 PSIA by 58 hours.
- Cell No. 4, with 20% KOH, was essentially voltage stabilized by 28 hours at 1.44 V and pressure stabilized by 40 hours at 23 PSIA.
- (4) <u>Discussion</u>. Filaments of 11 mil diameter do not impair overcharge ability at the C/20 rate in cells activated with 20% KOH.
- 2.1.1.2 Oxygen Electrode Effect in 4 AH Sealed Cell with Pillow

 Level Control. Pressure and level variation during 24 hour cycling has been determined for the same cell in two different conditions:

First - With a small oxygen electrode and a reduced

area of exposed plate surface (by initial filling to 50% level further reduced by level rise due to bellows action).

Second - With no oxygen electrode but with a relatively large area of exposed negative plate surface (by initial fill to only 10% level).

Following are test details and results:

a. Test Cell Characteristics (Cell No. 4).
Electrolyte = 20% KOH

Electrolyte Level at Seal = 100% immersion

under vacuum.

<u>Separator</u> = Filament plus cloth combination from previous test.

Ratio Charged Negative to Charged Positive = 1.3

Bellows: 4 mil polypropylene pillow, 2" x 1-7/8"

Oxygen Electrode. - An oxygen electrode,

5/8 x 3-1/4 x .015" was placed vertically next

to the element and opposite the plate edges

with the electrode plane being perpendicular

to the plane of the plates. This electrode was

grounded to the negative plate group by welded

connection inside the cell. A piece of 3 mil

EM476 non-woven material served to separate

the electrode from the plate edges and to supply

its inner face with electrolyte by wicking action.

A piece of 34 mil polyethylene "toy bag" netting with diamond axes 1/4" x 1/4" long was placed between the outer face and the container wall. The ratio of positive active plate area to outer oxygen electrode area was 22 to 1.

b. Test Cycle. -

Charge 22 hours at 0.25 ampere (1.37 C).

Discharge 2 hours at 1.40 amperes (0.7 C).

c. <u>Test Results</u>. - Figure 3 shows the variation of cell pressure and electrolyte level over a 24 hour period. The pressure developed in both cells remained essentially equivalent despite the considerable difference in plate flooding as outlined in the following table.

	<u>Electro</u> <u>Le</u> vel Var	
Period	Cell Without Electrode	Cell With Oxygen Electrode
Pressure buildup during charge	42% to 18%	100% to 46%
Pressure decline during discharge	18% to 23%	46% to 74%
Pressure decline during early charge	23% to 28%	74% to 94%

The difference is attributed to enhancement of oxygen recombination rate by the auxiliary electrode.

2.1.1.3 <u>Effect of Separator and Electrolyte Content on Bellows</u>Requirements. - The full range of liquid level control by action of a bellows

device containing trapped air can be accomplished in 3 different ways: chiefly by expansion; chiefly by compression; or by a combination of the two functions.

The mode of action in a sealed cell is dependent on three factors:

- a. Initial level or quantity of electrolyte in the cell <u>before</u> sealing.
- b. Initial pressure in the cell adjusted at the moment of sealing the cell by pressurizing, evacuating, or leaving the cell at atmospheric pressure.
- c. Quantity of air trapped in the bellows device.

For a specific bellows and for each of three different separators, the goal of this test has been a family of characteristic curves showing the liquid level versus cell pressure relationship at selected values of electrolyte content for adjusting test cells to operate within a reasonable range of pressures with a desired range of level control.

Following are test details and test results.

- a. <u>Test Cells</u>. Three 6 AH sintered plate cells were equipped with two rectangular, 4 mil polypropylene pillows, each 2 x 1-7/8 and each containing 1.5 cc of trapped air. The three separator materials used were:
 - (1) Separator A Single layer, Teflon monofilament screen, AGB9-60-250, 10 mils thick.

- (2) Separator B Double layer, nylon monofilament cloth,

 AGB8-M, 4 mils thick.
- (3) Separator C Three 11 mil polypropylene monofilament strands wrapped vertically around each positive plate and covered by Separator B.

The electrolyte was 20% KOH in all cells.

b. <u>Test Procedure</u>. -

- (1) Adjust electrolyte content of the vented cell to the desired starting level at atmospheric pressure.
- (2) Gradually evacuate the cell and note pressure at which the liquid level reaches the plate tops (100% immersion).
- (3) Vent the cell to atmospheric pressure and then admit compressed air. Raise cell pressure gradually and note the electrolyte level at definite steps. Continue until the liquid level has dropped to bottom of the plates or until a pressure of 60 PSIG has been reached.
- (4) Vent the cell to atmospheric pressure, adjust liquid level to the next selected height and repeat 2 and 3.
- (5) Repeat 2, 3 and 4 until the desired range of levels and pressures has been covered.
- c. <u>Test Results</u>. Figure 4 shows three sets of cell pressure vs liquid level curves, one set for each separator. Starting electrolyte levels cover the range 40% to 100% plate immersion. Imposed pressures range from 7 PSIA to 75 PSIA.

Several points are significant:

- (1) The bellows system consisting of two polypropylene pillows is more sensitive to expansion by pressure decline from atmospheric pressure than to compression by pressures in excess of atmospheric.
- (2) With Separator A (Teflon screen) the plate pack has a void volume for electrolyte that cannot be drained completely by this particular bellows from a starting level of 100% immersion.
- (3) Separator B (double layer nylon cloth) requires by far the least volume change and the lowest pressure range for complete flooding and draining of the cell pack.
- (4) The addition of filaments to Separator B reduces its pressure level characteristics to a condition even less favorable than that of Separator A.
- (5) Separator B allows an initially flooded cell to be drained by bellows action at an imposed pressure of only 2-1/2 absolute atmospheres. At 40% plate immersion, the lowest starting level used in this test series, Separator A requires a pressure greater than 2 absolute atmospheres to drain and Separator C needs more than 3 absolute atmospheres to drain.

- (6) The curves for Separator B indicate a reasonably practical match between the drain requirements of the element and the volume change capabilities of the bellows even where the cell is sealed in the flooded condition at atmospheric pressure. The curves for Separator A and C indicate a mismatch for starting levels above 40% plate immersion.
- d. <u>Discussion</u>. It is clear that initial electrolyte quantity is an important cell parameter to be evaluated for each particular combination of separator and bellows.

In this development, where a study of parameters such as the separator system can involve widely different degrees of bellows action, it becomes impractical from the standpoint of time and expense to purchase an equally wide supply of metal bellows to match each system. Fortunately, the capability has been developed for designing and fabricating plastic pillow type bellows to any degree of action required.

2.1.2 <u>Bellows Evaluation and Calibration</u>. -

2.1.2.1 <u>Development of Multi-Unit Plastic Bellows</u>. - Of all the plastic materials tested in the Second Quarter, the 4 mil polypropylene film, Olefane A-25, from Avisun Corporation, Philadelphia, Pennsylvania seemed the most promising as substitute for metal bellows. It proved resistant to KOH attack and easily heat sealable into "pillows" which displayed good volume

change properties. It also gave excellent flex life potential (6000 flexing cycles in 25% KOH at room temperature without failure). Accordingly, this film was adopted as the material of choice for plastic bellows development.

The plastic pillows used in the cell parameter study have been in the form of one or two large rectangular units operating in a chamber of ample volume and not always conforming precisely to the chamber shape. For example, in the tests described in the previous section, the two rectangular pillows were folded to fit into the cylindrical bellows chamber of the 6 AH test cell.

It should be recalled that a cylindrical chamber was selected to conform to cylindrical metal bellows and to provide access to the chamber by a threaded end cap.

Since efforts to locate a small ready-made sealed plastic bellows were unsuccessful, it was decided to make a bellows equivalent by grouping together individual, heat-sealed, round polypropylene film pillows. Such grouping, while functioning as a conventional single cavity convoluted bellows, offers the versatility of discreet units which may be altered in number to change the bellows capability. Also, a leak in one unit will not cause complete bellows failure as could happen in conventional bellows.

A pillow O.D. of 1-1/8" was chosen to utilize most completely the chamber diameter of 1-1/4" without binding. A 1/8" wide heat seal was used at first and later reduced to 1/16" to provide more volume for trapped air as the reliability of the seal was demonstrated.

Due to the relatively small size of the round pillow, accurate addition of air by hypodermic needle as done with the large rectangular pillow proved difficult and slow. This problem was solved by merely encapsulating in each pillow a small crushable pill of expanded polystyrene cut to exact dimensions. The pill assures a reproducible volume of trapped air from pillow to pillow and, after being crushed, leaves room for flexing.

Since pill thickness governs the initial spacing of the pillow wall and thus determines the volume of trapped air, a calibration test was run using pillows with initial wall spacings of 0.050", 0.060" and 0.070".

Each calibration was made by inserting 22 pillows, all of the same wall spacing, into the cylindrical bellows chamber and, after filling the chamber and an attached calibrated tube with water, measuring the volume changes over a range of imposed pressure above and below atmospheric. Figure 5 clearly indicates that the total volume change, the sum of the compression

and expansion volume changes, increases with pillow inside wall spacing in the range 0.050" to 0.070".

b. Flex Life Testing of Polypropylene Pillows. - A number of flex life tests in 20% KOH were run for circular pillows with both a 1/8" and 1/16" seal widths. This test has become both an evaluation test for new materials and a quality control test.

At room temperature, one batch of eleven 1/8" seal pillows ran 10,600 cycles without failure. In a batch of thirty-one 1/16" seal pillows, one leaked at 7700 cycles and the remainder ran 14,800 cycles without failure. The one failure was due to poor sealing techniques which were subsequently improved with better sealing tools and closer heat control.

A batch of eleven 1/8" seal pillows was given a sequence of flex tests: first at room temperature, then at 0° C and finally at 40° C. The test ran a total of 26,500 cycles without failure. The details are tabulated below:

Room Temperature (15 sec. cycle); ll sec. evacuation to 28" Hg and 4 sec. exhaust. No. of cycles = 7,700

O°C and (30°F) (35 sec. cycle): 30 sec. evacuation to 28"

Hg and 5 sec. exhaust. No. of cycles = 7,800

40°C (104°F) (10 sec. cycle): 7 sec. evacuation to 28"

Hg and 3 sec. exhaust. No. of cycles = 11,000

A batch of eleven 1/8" seal pillows made of the DuPont polyionomer, Surlyn A, was run through the temperature sequence flex test. No mechanical failure occurred but KOH was found inside all of the pillows.

A batch of twenty-two 1/16" seal rectangular polypropylene pillows ran 28,200 cycles at room temperature without failure. This pillow design was developed for the Task 2 Ag-Cd test cells.

2.1.2.2 <u>Calibration of Nickel and Polypropylene Bellows</u>. - Utilizing the same test chamber and technique as used in the individual pillow development, calibration tests were run at room temperature on all ten nickel bellows and on polypropylene bellows made up of six different numbers of pillow units - 25, 30, 35, 40, 45 and 50 units. The 35 unit polypropylene bellows was also calibrated at 0°C and 40°C. The results are shown in Figures 6 and 7.

Several points are worthy of discussion:

- a. Each calibration curve has two arms representing volume change. One is due to bellows expansion and the other to bellows compression.
- b. Both sizes of metal bellows divide their rated capacity equally between expansion and contraction. They both meet rated capacity but are limited by the manufacturer to a maximum pressure beyond which the cycle life is reduced.

lene film bellows directly affects the division of its action between expansion and contraction. This can be seen from the shape of the calibration curves in Figure 7. Calibration figures are tabulated below over the range 10 to 60 PSIA. They show the transition from 46% compressive action for a 25 unit bellows to 71% compressive action for a 50 unit bellows.

Number of Pillow	Total	Volume Ch Amount Compre	ssion	en 10 and 6 Amount D Expans	ue to sion
_Units	CC	CC	%%	cc	%
25	13	6	46	7	54
30	14.5	6.5	45	8	55
35	17	8	47	9	53
40	19.5	10	51	9.5	49
45	19	12	63	7	37
50	19	13.5	71	5.5	29

Note that while a 40 unit bellows acts almost equally between compression and expansion, the addition of more units increases compressive action but decreases expansive action by a corresponding amount. This characteristic of multi-unit plastic bellows thus offers the possibility of assembling a bellows "tailored" to the job.

d. Figure 6 shows almost identical shape and range for the calibration curves of the 35 unit plastic bellows at $0^{\circ}\mathrm{C}$

- and 40°C. At both temperatures total volume change between the low and high pressure extremes is inferior to that found at 25°C.
- e. In summary, a capability for producing "tailored" bellows action has been developed using inexpensive and easily obtained materials. Good performance and life equivalent to one year of 90 minute cycling orbits at the temperature extremes of this study may be reasonably expected. It now remains to incorporate this know-how into the evaluation program and into the final cells for delivery.

2.1.3 Evaluation Test Program. -

2.1.3.1 Modification of the Plan. - As originally outlined in Tables (3) VII and VIII of the Second Quarterly Report, the evaluation test schedule was built around fifteen 6 AH cells divided into three groups: one group of 6 cells to receive baseline and proof cycle tests, a group of three to function as control cells and another group of six to stand in "reserve" for final assembly and final cycle testing under optimum conditions.

The initial group of six was designed to evaluate double layer, coarse weave nylon cloth and single layer teflon screen which had been selected in the second quarter as the most promising separator materials.

The three control cells were to function as conventional, limited electrolyte, sealed cells with one containing Pormax separators and the other two the cloth and screen materials.

Final assembly of the "reserve" or "optimized" group was dependent on performance of the initial group.

As mentioned earlier, the basic evaluation schedule is being followed but the spectrum of parameters to be adjusted for optimizing performance has been extended. Table I presents an outline of the plan for continuing the parameter study.

This change in the program plan has been made in the light of circumstances listed below and is expected to broaden the scope of study without undue expenditure of time, effort or materials.

- a. Cells No. 1 through 4 were assembled with an accordian wrap separator system of one layer of 4 mil nylon cloth adjacent the positive plates plus one layer of teflon screen against the negative plates to evaluate the combination of materials.
- b. Cells No. 10 through 15 were assembled with an accordian wrap of two layers of nylon cloth which appeared to be an optimum system from previous work in the Second Quarter. Cells No. 14 and 15 were also assembled using smooth monofilament, 11 mil diameter polypropylene strands tied vertically around each negative plate as a contrast to the cells already tested with the strands wrapped around the positive plates. The strands should aid in the removal of gases trapped in the cloth meshes and facilitate rapid and efficient drainage of electrolyte.

TABLE I

PARAMETER STUDY OUTLINE FOR EVALUATION IN FIFTEEN SEALED NICA CELLS

			(1) Basic Electrolyte	(1) Imposed Pressure	Aux.	
Cell No.	Separator System	Bellows	Content (Pre-Seal Level)	At Seal (Post-Seal Level)	02 Elec- trode	Principal Parameters Evaluated
H	<pre>lL, 4 Mil, Nylon Cloth lL, 10 Mil, Teflon Screen</pre>	20 cc Metal	5 0%	Evacuate To Flood	Yes	Separator and Bellows
2	<pre>LL, 4 Mil, Nylon Cloth LL, 10 Mil, Teflon Screen</pre>	10 cc Metal	5 0%	Evacuate To Flood	Yes	Separator and Bellows
3	<pre>LL, # Mil, Nylon Cloth LL, 10 Mil, Teflon Screen</pre>	40 pillows	5.0%	Evacuate To Flood	Yes	Separator and Bellows
±	<pre>1L, 4 Mil, Nylon Cloth 1L, 10 Mil, Teflon Screen</pre>	50 Pillows	20%	Evacuate To Flood	Yes	Bellows
5	lL, 10 Mil Teflon Screen	20 cc Metal	20%	Evacuate To Flood	Yes	Separator and Bellows
9	lL, 10 Mil Teflon Screen	10 cc Metal	20%	Evacuate To Flood	Yes	Separator and Bellows
7	lL, 10 Mil Teflon Screen	μ0 Pillows	20%	Evacuate To Flood	Yes	Separator and Bellows
8	lL, 10 Mil Teflon Screen	50 Pillows	5 0%	Evacuate To Flood	Yes	Bellows
6	Pormax	None	Forced Drain	Atmos.	Yes	Control
10	2L, 4 Mil, Nylon Cloth (8 Mils Total Thickness)	10 cc Metal	%05	Evacuate To Flood	Yes	Separator and Bellows

TABLE I

PARAMETER STUDY OUTLINE FOR EVALUATION IN FIFTEEN SEALED Ni-Cd CELLS (Continued)

Cell No.	Separator System	Bellows	(1) Basic Electrolyte Content (Pre-Seal Level)	(1) Imposed Pressure At Seal (Post-Seal Level)	Aux. 02 Elec- trode	Principal Parameters Evaluated
11	2L, 4 Mil, Nylon Cloth (8 Mils Total Thickness)	Pillows	%05	Evacuate To Flood	Yes	Separator and Bellows
12	<pre>2L, # Mil, Nylon Cloth (8 Mils Total Thickness)</pre>	10 cc Metal	20%	Evacuate To Flood	No	O ₂ Aux. Elect. Effect
13	2L, 4 Mil, Nylon Cloth (8 Mils Total Thickness)	Pillows	20%	Evacuate To Flood	Ño	O ₂ Aux. Elect. Effect
14	Same Plus ll Mil Strands on Negative Plates	20 cc Metal	20%	Evacuate To Flood	No	Separator and Aux. Elect.
15	Same plus ll Mil Strands on Negative Plates	20 cc Metal	20%	Evacuate To Flood	Yes	Separator and Aux. Elect.

These parameters will be adjusted if required during the course of the program. (T)NOTE:

- c. During the sealed overcharge capability tests, described later in this report, the two limited electrolyte "control" cells with open weave separator (cells No. 4 and No. 8) were found incapable of continued sealed operation. It was decided to convert this pair into evaluation cells for plastic pillows. Cell No. 9 with the highly absorbent Pormax separator remains as a "control" cell with a non-changing electrolyte level.
- d. To check oxygen electrode effectiveness by direct comparison in 4 basically equivalent cells, two cells were assigned for tests with the oxygen electrodes and two without
- e. Three departures from the original test plan are to be followed:
 - (1) The orbit cycle range will be 24 hours to 8 hours instead of 24 hours to 2 hours. This is in accord with verbal agreement during the NASA-ESB conference of February 15, 1965 at Raleigh, N. C.
 - (2) The high temperature limit is lowered from 50°C to 40°C per verbal agreement during the same conference.
 - (3) Tests at 0°C and 40°C will be deferred until satisfactory performance at 25°C has been attained.

- 2.1.3.2 <u>Progress in the Evaluation Test Schedule</u>. Tests, involving cells 1 through 9, have progressed through the following stages:
 - a. Measurement of bellows action with 4 different separator systems.
 - b. Manual baseline tests
 - (1) Open cell discharge capacity at various rages and electrolyte levels.
 - (2) Sealed cell overcharge capability.
 - c. Proof cycle tests
 - (1) Initial performance in 24 hour orbit cycle.

Details of these tests are outlined below:

- a. <u>Measurement of Bellows Action</u>. This was a calibration test with bellows functioning by pressures imposed on the actual test cells.
 Parameters covered were:
 - (1) Bellows Type 20 cc nickel, 10 cc nickel and 31 unit polypropylene.
 - (2) Separator 1 layer teflon screen alone (Separator Type "A").
 - 2 layers nylon cloth (Separator
 Type "B").
 - 2 layers nylon cloth with filament
 on positive plate (Separator Type
 "C").
 - l layer nylon cloth plus 1 layer
 teflon screen (Separator Type "D").

- (3) Mode of bellows action Manually increasing and decreasing pressure in the cell.
- (4) Electrolyte Content 50% level at atmospheric pressure.

<u>Test Results</u>. - Figures 8, 9, 10 and 11 show the following points of interest:

The <u>10 cc bellows</u> can drain only one cell (Separator Type "B") without being compressed beyond the manufacturers recommended limit.

The <u>31 unit polypropylene bellows</u> cannot drain Separator Types "A" and "D" at practical cell pressures.

Bellows action shows a <u>hysteresis effect</u> for all separators.

This effect may be due to the time factor of approximately

1 minute between pressure increments.

Discussion: This test illustrates both adequate and inadequate bellows action. The 20 cc bellows provides complete flooding and drain at reasonable pressure limits for all separator types.

The 10 cc bellows is suited for operation with Separator "B" only. The polypropylene bellows can be made to provide adequate drainage by increasing the number of its units.

The calibration curves of Figure 7 indicate the use of 40 units.

b. Manual Baseline Tests. -

(1) Open Cell Discharge Capacity. - Tests were run on all cells at discharge rates of C/5, C/2 and C/l and at electrolyte

levels of 100%, 50% and 10% immersion. Typical performance for each of the three separators is plotted in Figures 12, 13 and 14.

A capacity drop with reduced electrolyte level was observed at all three discharge rates. Cell control No. 9 with Pormax separators also showed a sensitivity to level, especially between 100% and 50% plate immersion.

(2) Sealed Cell Overcharge Capability. -

Preparation of Cells for Sealed Operation. - At the end of the preceding test all nine cells were flooded and discharged to zero volts at the C/5 rate and continued into reverse charge to prepare the negative groups for sealed operation. The reverse charge for the three "control" cells was done with the cells drained and inverted to put them in the conventional "forced drain" condition. The extent of reverse charge assured a minimum value of 1.35 for the ratio of charged negative active material to charged positive active material for all nine cells. The control cells were sealed at atmospheric pressure without evacuation. Their separator systems are as follows:

<u>Cell No.</u>		Separator
6-4		lL nylon cloth + lL teflon screen
6-8	-	lL teflon screen
6-9	-	Pormax

The six baseline test cells were sealed after initial electrolyte level adjustment to 50% immersion followed by evacuation. Cell make-up and cell conditions at sealing are listed below.

			Adjusted Cell
Cell No.*	<u>Separator</u>	<u>Bellows</u>	Condition at Seal
6-1	<pre>lL Nylon Cloth + lL Teflon Screen</pre>	20 cc nickel	100% level at 3.9 PSIA
6-2	lL Nylon Cloth + lL Teflon Screen	10 cc nickel	87% level at 1.5 PSIA**
6-3	<pre>lL Nylon Cloth + lL Teflon Screen</pre>	31 - pillow	100% level at 8.3 PSIA
6-5	lL Teflon Screen	20 cc nickel	100% level at 4.9 PSIA
6-6	lL Teflon Screen	10 cc nickel	90% level at 2.9 PSIA**
6-7	lL Teflon Screen	31 - pillow	100% level at 8.3 PSIA
NOTE: *	All cells, including	the control group,	, have auxiliary
	oxygen recombination	electrodes. The m	ratio of active
	auxiliary electrode f	ace area to active	e positive plate
	face area is 1/6.		

** The 10 cc bellows was unable to completely flood the element, even at low pressures.

Overcharge Test. - Cell voltage, pressure and electrolyte level were monitored for all nine cells over the test sequence: charge at C/20, overcharge at C/20, C/10 and C/5; open-circuit stand and discharge at 2.0 amperes. Cell conditions at the end of each step are listed in Table 2. Patterns of response for all cells over the entire sequence are plotted in Figures 15, 16, 17 and 18.

TABLE II

FINAL DATA AT EACH STAGE DURING OVERCHARGE CAPABILITY
TEST OF NINE 6 AH SEALED Ni-Cd CELLS - SIX CELLS WITH BELLOWS
AND THREE WITH LIMITED ELECTROLYTE TO ACT AS "CONTROL" CELLS

		Test Cell Groups								
		Control Group			Nylon-Teflon			Teflon Sep.		
Test Step		(No Bellows)			Sep. Group			Group		
	4	8	9	1	2	3	5	6	7_	
Cell Number *	NY-Tef	Tet.	Pormax	20cc B	Tucc B	3T-B	20cc B	TOCC B	31-P	
Charge at C20 Hr	s. 26	41	41	41	41	41	41	41	41	
Charge at C20 H		1.56	1.40	1.40	1.40	1.40		1.41	1.46	
PSI		1.30	35	18-1/2	28	24		21	20	
Level				62	60	64	60	62	57	
Overchg. @ C/10 Hr	i i	1/2	24	24	24	24	24	24	1/2	
_ V	i	1.72	1.40	1.40	1.45	1.41	1	1.48	1.55	
Pres		25	54	23	43	35	23	25	22	
Leve	1			42	45	47	47	52	52	
Overcharge @ C/5 Hr			4	ц	0.1	ц	0.1	0.1		
V	1		1.42	1.44	1.55	1.43	1	1.64		
Pres	s		83	30		46				
Leve	1			23	~-	39				
		00.3/6								
Open Circuit Stand H		29-1/2	•	2	6	2	6	6	29-1/2	
V Dm o o		1.33	1.33	1.34	1.34 9.8		1.34	1.34	1.33	
Pres Leve			44 ~-	12.7 87	9.0	73	11.8	8.8	10.8	
reve		 	 	07		/				
Dischg. @ 2.0 Amps										
	s 1.13	2.28	2.62	2.67	2.48	2.62	2.61	2.65	2.85	
/	1.00	1.00	1.00	1.00	1.00		1.00	1.00	1.00	
Pres	1	21		7.3	6.9	13.2		5.4	9.9	
Leve	1			95	86	96	90	84	98	

^(*) NY-Tef = Sep. of 1 layer nylon cloth plus 1 layer teflon screen.

Tef. = Sec. of 1 layer teflon screen only.

Pormax = Sep. of microporous PVC sheet.

20 cc B = Metal bellows of 20 cc capacity.

10 cc B = Metal bellows of 10 cc capacity.

31-P = Polypropylene bellows of 31 pillow units.

Note on cell numbers: To distinguish between the 4 AH and 6 AH cells, the latter are identified on graphs by the pre-fix numeral "6". For example, cell No. 4 in this table appears as cell No. 6-4 in Figure 15.

Discussion of Test Results. -

(a) Figure 15 and Table 2 show the inability of "control" cells

No. 4 and 8 to tolerate overcharge at the C/10 rate. The

open structure and the hydrophobic nature of the coarse

weave separator material may prevent the retention of an

adequate supply of electrolyte. These two cells, as mentioned

earlier, have been converted into pillow evaluation cells.

The remaining control cell, containing Pormax, showed excellent overcharge capability, even at the C/5 rate.

- (b) Bellows action for both sizes of metal bellows and for the plastic bellows was synchronous with pressure variation throughout the entire 74 hour sequence of events.
- (c) The cells with cloth-screen combination separator (abbreviated NY-TEF) seem able to withstand higher overcharge rates than those with screen alone (abbreviated TEF) at comparable levels of electrolyte. For example, all three NY-TEF cells withstood 24 hours at C/10 rate and two ran the 4 hours at C/5. Only two of the TEF cells ran full time at C/10 and none could take C/5.
- (d) The electrolyte level vs cell pressure relationship in this test does not correspond to that displayed in the calibration curves of Figures 10 and 11. For example,

in Table 2, at the C/5 rate, a level of 23% is shown for a pressure of 30 PSIA in cell No. 1 (20 cc bellows and NY-TEF Sep.). The calibration curve for this cell in Figure 11 indicates a level from 0 to 5% for 30 PSIA. This difference is attributed to the presence of cell gas which is being evolved vigorously at the C/5 rate. Erratic points in the pressure and level plots are due to the dislodgement of trapped gas with resultant level drop and corresponding pressure dip.

c. Proof Cycle Tests. - Following the experience gained in the baseline tests and in accord with the outline for parameter study, the following changes in cell make up were made. Former control cells No. 4 and 8 were equipped with polypropylene bellows of 50 pillow units. Cells No. 3 and 7 were modified from 31 pillow units to 40 units per bellows system. All 8 bellows equipped cells and the control cell were placed on manual 24 hour orbit cycling for rate adjustment to permit automatic unattended cycling. The initial test regime is as follows:

Charge 22 hours at 375 mA (1.37 C input)

Discharge 2 hours at 2.1 amps (0.7 C output)

Figures 19 through 22 show plots of the cell pressure and
level variation over one complete cycle for the eight cells
paired according to bellows type.

A number of points should be noted:

- (1) As found in the preceding baseline tests, bellows
 response to cell pressure changes occurred in a synchronous
 manner.
- (2) All four graphs show double points for electrolyte level in the last third of the charge period when gassing is most vigorous. This fluctuation indicates the degree of gas entrapment occurring with these two separator systems.
- (3) Both pressure and level variation in the last third of charge is noticeably more erratic for the all teflon screen separator than is found with the cloth-screen combination. This may indicate a channeling action inside the cloth which reduces entrapment by the screen horizontal cross members.
- (4) At the end of charge, the prompt pressure decline and level build up indicate both good recombination rate and good bellows response. By the end of discharge, the recovery of liquid level is nearly complete. This favorable interaction occurs with both separators and both types of bellows.
- (5) It should be noted that this evidence of good recombination has occurred at relatively high liquid levels and is in contrast to the poor performance obtained in the second quarter with much lower levels.

Since this improvement may be due in large measure to the

auxiliary electrode, the planned tests for evaluation of this electrode by direct comparison are of considerable interest.

Twenty-four hour cycle tests are nearing completion at room temperature and seemingly reliable, unattended cycling has been observed at charge rates near C/6.

Further optimization of the plastic bellows assembly will precede the tests at $0^{\circ}C$ and $40^{\circ}C$.

- 2.2 <u>Task 2 Feasibility of Bellows Controlled Electrolyte Level</u>

 <u>In Sealed Cd/KOH/AgO and Zn/KOH/AgO Cell. -</u>
- 2.2.1 <u>General Cell Design</u>. An 8 AH sealed silver cadmium cell with plastic bellows has been designed and 24 are under production.

The cell was designed around the transparent RMD-4511 polystyrene cell case for which ESB purchased tooling on Contract NAS5-1607. A general outline of the cell assembly is shown in Figure 23.

The bellows will be composed of rectangular heat sealed polypropylene film pillows held together in a lucite cage and installed in the bottom of the cell. Figure 24 illustrates the basic pillow unit, the heat sealing die and the cage. This bellows is a rectangular version of the round unit plastic bellows developed in Task 1.

A number of points should be considered regarding this experimental cell design:

a. The plate pack does not occupy the entire space

in the cell jar above the bellows and below the cover. Plastic shims occupy the unused space to maintain pack tightness and reduce the volume of free electrolyte to be handled by the bellows.

- b. The usual space is left above the plate tops for separator extension. This is utilized to enhance oxygen recombination by running the auxiliary electrodes all the way up for maximum surface area for reaction sites. The ratio of oxygen electrode exposed face area to the positive plate face area in this cell is 1/3.
- c. A third terminal is provided for the oxygen electrode.

 This permits a study of performance with and without the auxiliary in electrical connection to the negative cell post. Measurement of recombination branch current may be done with an ammeter in this external connection.
- d. The total cell jar <u>height</u> below the cover is occupied in the following proportion:

Bellows = 24%

Plate Pack = 59%

Void space above and below plate pack = 17%.

In comparing the actual <u>volumes</u> occupied, the proportion becomes:

Bellows volume = 30%

Plate pack volume = 55%

Void volume = 15%

2.2.2 <u>Plate Pack Design</u>. - The 24 cells are divided equally amont four separator systems as shown in Figure 25. Note that the Borden C-3 material is employed by U-fold assembly, in one case on the positive plates and in the other on the negative plates. The proprietary material, RC-901, is used as a heat sealed bag.

The plate pack composition of 4 positive and 5 negative plates uses full plate thickness on the outer negatives to provide adequate supply of negative active material.

The oxygen electrodes are supplied with electrolyte by the strong wicking properties of EM-476 separating it from the outer negatives. Net spacers are used to separate the outer face of the electrodes from the inner surfaces of the cell jar.

2.3 <u>Task 3 - Feasibility of Bellows Actuated Charge Cut-off</u>. - This approach to charge and level control is being made on the basis of the Task 2 cell design.

Figure 26 illustrates the design now being negotiated with the bellows supplier for the bellows actuated mechanical switch combination. The basic switch being considered is the Type AT1-1 subminiature unit made by Metals and Controls, Inc., a division of Texas Instruments, Inc.

The plastic bellows should be utilizable in combination with a pressure actuated switch. Negotiations are under way with Texas

Instruments, Inc. for use of a switch similar to their Types 2PS and 3PS

The metal bellows under design for mechanical switch actuation is limited in travel by the use of restraining springs. To compensate for this lack of volume change, it seems quite feasible to use plastic pillows in the same bellows chamber. In this way, charge control should be obtainable through both switching action and liquid level variation.

3. NEW TECHNOLOGY

No new technology has been introduced into the program during the third quarter.

4. PROGRAM FOR NEXT QUARTER

- 4.1 <u>Task 1.</u> The test program will be completed using the optimized 6 ampere hour Ni-Cd sealed cells equipped with both metal and plastic bellows. Continuous cycling at the shortest orbit will be attempted, with the 8 hour orbit as a goal. Tests at 0°C and 40°C will be run.
- 4.2 <u>Task 2</u>. Complete the assembly of 24 sealed Ag-Cd test cells with plastic bellows level control, oxygen electrodes and four different separator systems. Initiate the test program. Optimize electrolyte content and complete the test program at the shortest possible orbit.
- 4.3 <u>Task 3.</u> Complete design, procurement and installation of the bellows-switch combination for both mechanical and pressure switches.

 Utilize plastic bellows in combination with metal bellows where indicated.

 Evaluate in the test cells of Task 2.

5. CONCLUSIONS AND RECOMMENDATIONS

- 5.1 Adequate level control using both metal and plastic bellows has been demonstrated in 6 AH sealed Ni-Cd cells with oxygen electrodes in a 24 hour orbit. Electrolyte level has varied from full flood to below 40% immersion.
- 5.2 Versatility in fabrication and operation of a plastic pillow type bellows has been developed. By adjustment of the number of "pillow" units in a given bellows chamber, the bellows action can be changed in character from mostly compression to chiefly expansion or it can be equally divided between the two.
- 5.3 Thin polypropylene film is the best plastic bellows material obtained. It is easily heat sealed and has excellent flexing properties. No failure was reached after flexing under 20% KOH for 26,500 cycles, of which 7,700 cycles were run at room temperature, 7,800 cycles at 0°C and 11,000 cycles at 40°C. Rectangular pillows were flexed 28,000 cycles at room temperature without failure.
- 5.4 The utilization of pressure sensitive and mechanical switches with bellows appears promising. A combination of metal and plastic bellows is worthy of trial.

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APPENDIX A

Correction of error in Second Quarterly Report on Page 45 in Table VII, "Design Factors for 15 - 6 Ampere-Hour Cd/KOH/NiOOH Sintered Plate Test Cells for Evaluation of Bellows Action".

INCORRECT

PLATES

Positive - 6 plates @ 1-3/4 X 1-3/4 X 0.050"

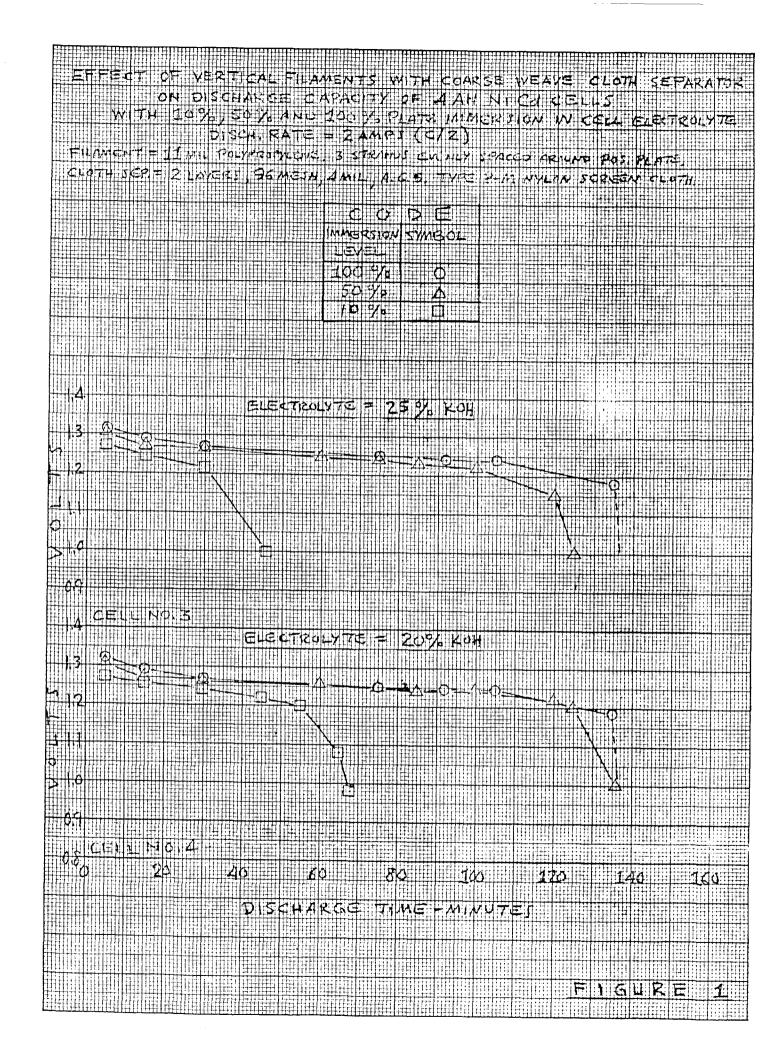
Negative - 7 plates @ 1-3/4 X 1-3/4 X 0.050"

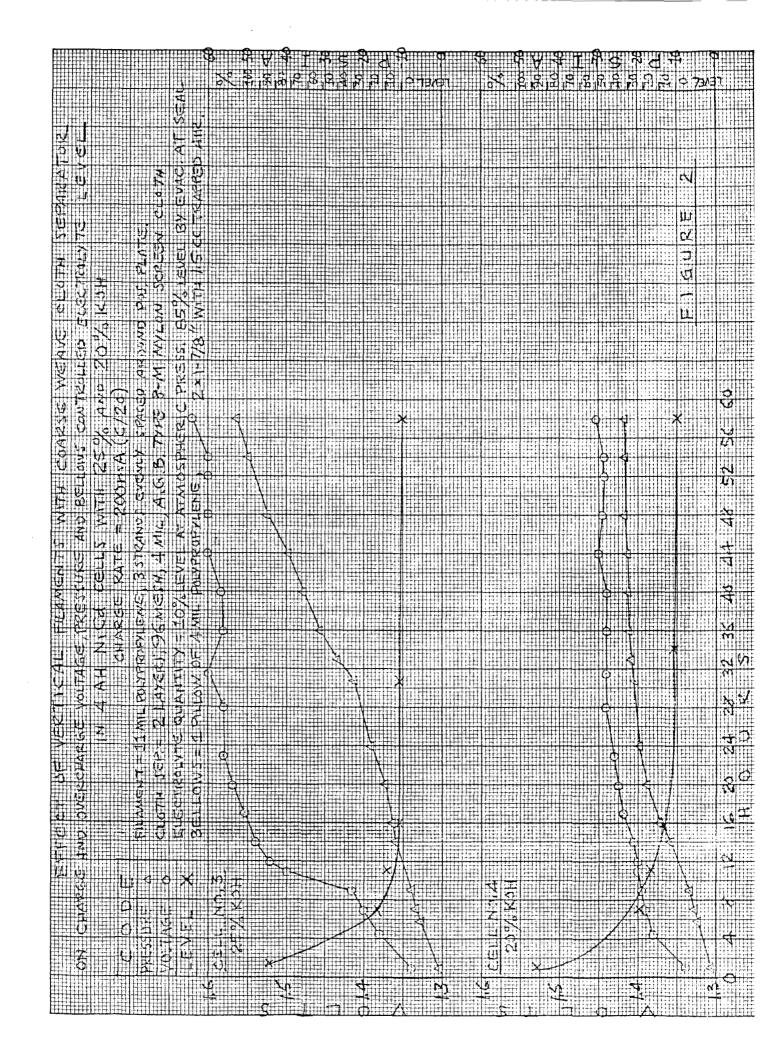
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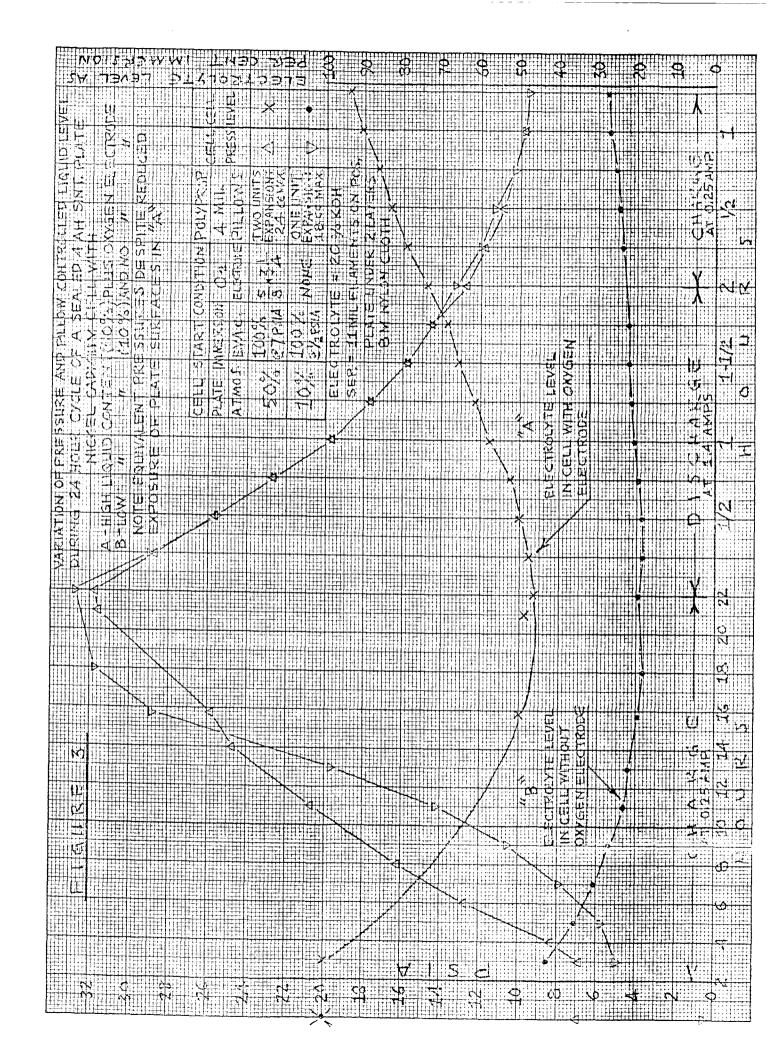
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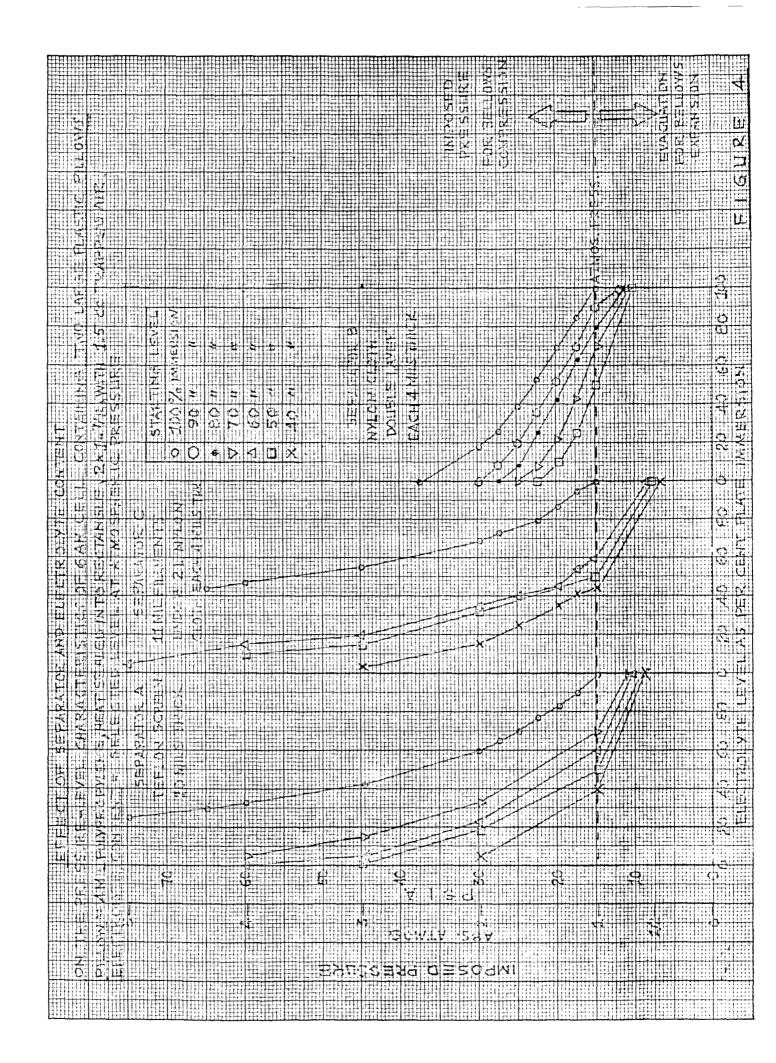
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Negative - 7 plates @ 1-3/4 X 3-1/4 X 0.050"









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**DE IN U.S.A.

KEUFFEL & ESSER CO.

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MOE 10 X 10 THE CENTIMETER 46 1513
IS X 25 CM. MADE IN U.S.A. KEUFFEL & ESSER CO.

SG WEEH WHON CLOTH # 11 JO MILLS INWEST TEFFLOW SCREEN

PRESSURE

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FIGURE RESSLIKE TE NICU SEALSD CELLS 17 ED (PR) KED DEX CTROUYTE LEVEL WITH TAIPES THREE DIFFERENT BELLOWS IN & AH SINTERE

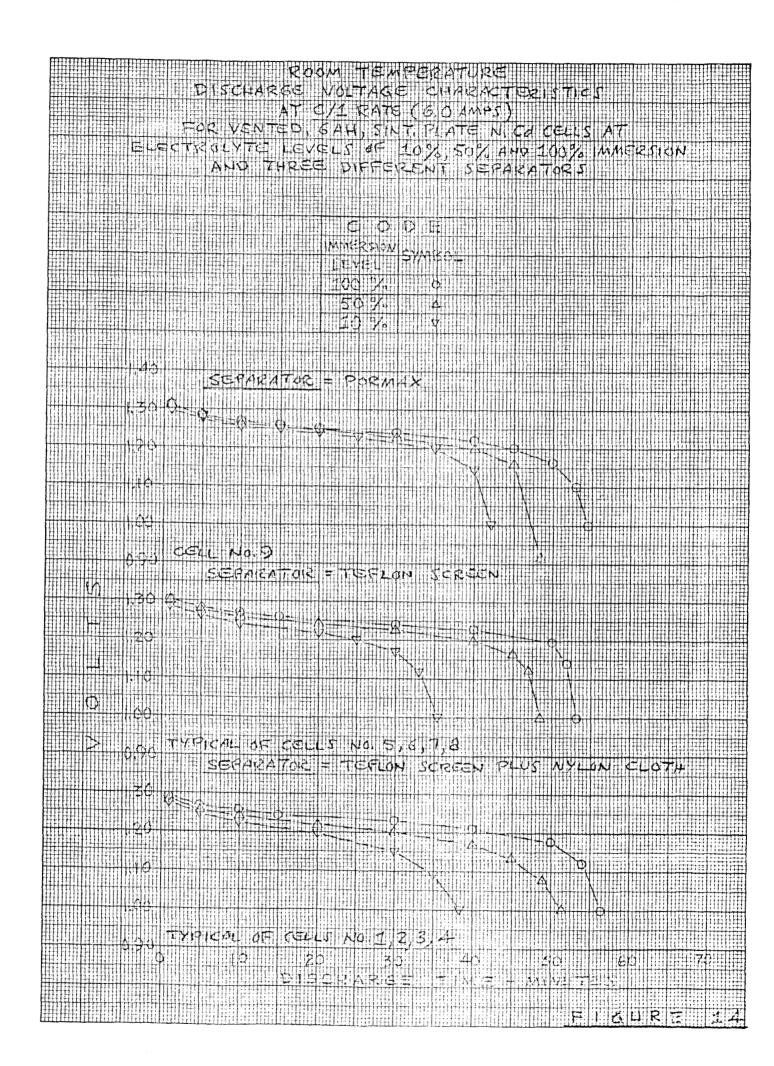
SO = DECREASING PRESSURE

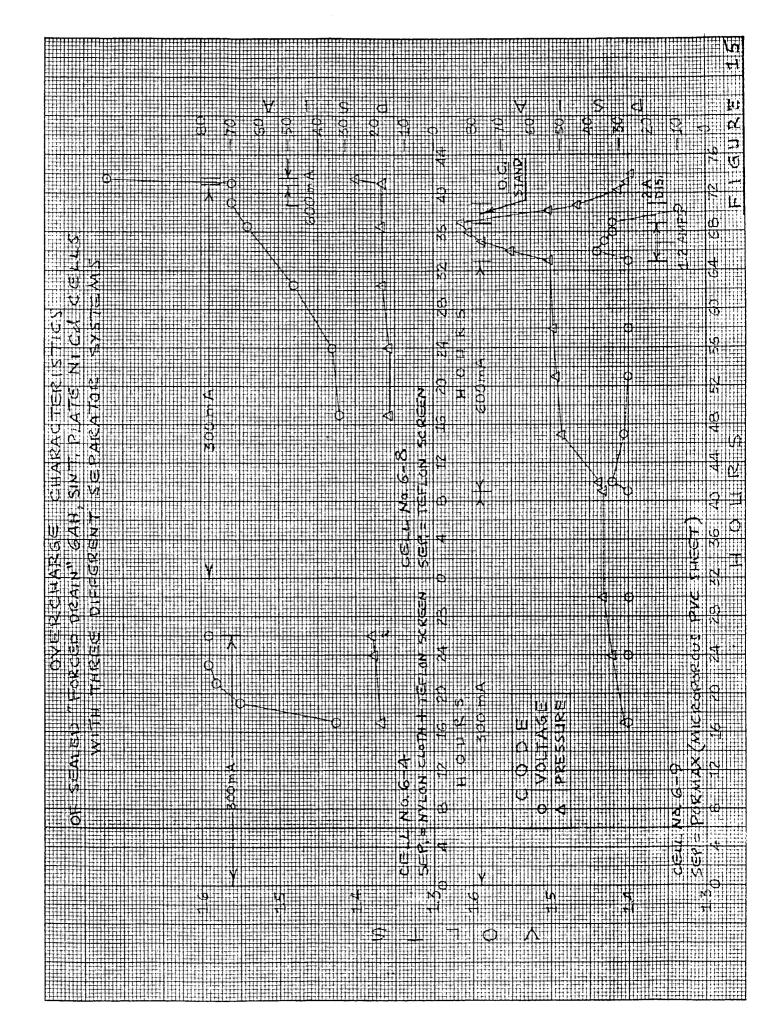
A = INCREASING PRESSURE

(D = STARTING FREESTROWTE LEYEL AT

(C) = MAX, PRESSURE FOR BEST METALS EFF OME THREE SEPARATOR D U FOR FIGURE

DISCHARGE VOLTAGE CHARACTERISTICS
AT C/S RATE (1.2 AMPS)
FOR VENTED, GAH, SINT, PLATE IN CALCELLS AT
ELECTROLYTE LEVELS OF 10%, 50% AND 100% IMMERSION
AND THREE PIFFECENT SEPARATORS
IMMERSION SYMBOL
TOO % O
50 %
140 SEPARATOR + PORMAX
Haoli Haoli Halling and the second a
0.90 CEAL NO.9 SERARATOR = TEFLON SCREEN
Un Bolling Company Company
O LOS TYPICAL OF FELLS WO 5, G, 7, 8
TO 100 P A D D TYPICAL OF FELLS WO 5, G, 77, 8 SEPARATOR = TEFLON SCIPCEN TRUS NYLON ELDTH
O 100 A PO TYPICAL OF FELLS NO. 5, A, 7, 8 SEPARATOR = TEFLON SCIPCEN PLUS NYLON CLOTA 30 60 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0
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AND ELECTROLY SETTLOWS OV SWITH Z DIF	×dó / V	X	
PRESSURE OFCE METAL NICCICELL CHARGE SE SE SE SAMPS			X
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CTROLYTE VOLUME DUG TO REA 74 HOUR CYCLE	A PRESTURE X LEVEL G			48: 32: 56: 60: 64: 63: 72: 76: 0 5: 17: C
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KON 10 X 10 THE CENTIMETER 46 1513

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H4 18 x 25 CM. NEUFFEL & ESSER CO.

ROOM TEMP. E 22 HOURS AT 380 m A (1,4C) CHBELEDWS OF THINCH DIAM ZAHOUR ORBIT CYCLE AT ROOM TEMP COMBINATION NYLON CLOTH-TEFLON SCREEN DISCH 2 HOU CELL NO. BELLOWS PRESS 6-1 20 CC A 6-2 10 CC A CTION OF 10 LO AND 20 LE NICKE!

DURING ZAHODR ORBIT REIT REGIME: CHARGE FIGURE

HOE 10 X 10 TO THE CENTIMETER 46 1513

NAME 18 X 25 CM

KEUFFEL & ESSER CO.

Z. Z. APARES (OL 77.C.) ND 2000 NGKEL SELLOWS OF TINCH DIA 24 HOUS CRAIT GYOLS AT ROUL TOUR GD SAH SINT PLATE NEGLOCILE WITH SLE LAYER HE FION SOREEN STRAKATION BOPA (CHARGE 22 HOURT AT 3월 DISCH, 2 HOURT AT Z. 4 Δ CEUL NO. BELLOWER P 6-5-20 CC 6-6-10 CC REGIMEN CHARGE Z V STALED FINSLE E HOCH AS Ö CTECK = 41 20 FIGURE

10 X 10 TO THE CENTIMETER 46 1513 MADE IN X 25 CM. MADE IN U. S.A. KEUFFEL & ESSER CO.

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KOE 10 X 10 THE CENTIMETER 46 1513 RATE 18 X 25 CM. KEUFFEL & ESSER CO.

ORENT RESIDENT BOUND POLYSON BELOWS

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COLUM ACTION OF M A A H H FIGUR

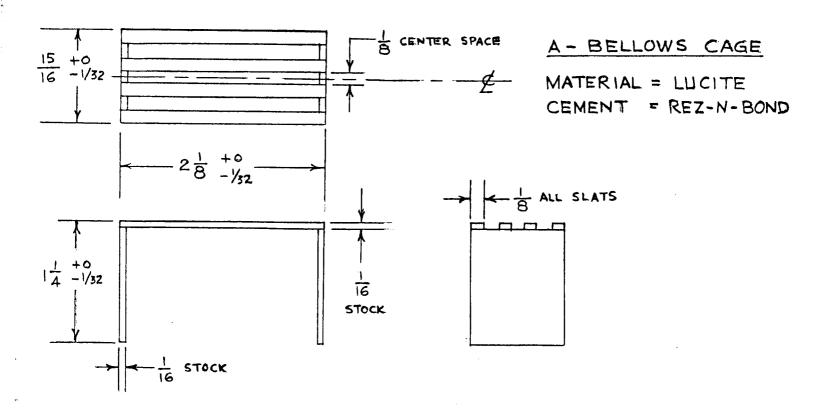
CHKD. BY DAT	EOR	BAH SEAL	> JOINT AROUN' 5 PSI INTERNA > EPOXY, ESB-1 72°±5° FOR	SESS ESB D POTTING AL PRESS, SS-300, T 8 TO 96	
3		9 8	10 11 12 14	J-253	11-JAR, 216-2001-3 21-POTTING COVER SK-83276-T2-1B 32-LEAD SLOT COVER SK-83276-T2-1D 41-CELL COVER SK-83276-T2-1A 52-CELL POST 150-10006-11 62-GASKET 150-10014-2 71-VENT HOLE COLLAR SK-83276-T2-1C 81-AUX, ELECT. POST 150-10006-9 91-GASKET 150-10014-8 102-PLASTIC SHIM SK-83276-T2-11 112-NET SPACER SK-83276-T2-11 12-AUX, ELECTRODE SK-83276-T2-4 131-BELLOWS CAGE SK-83276-T2-10A 14 SPACE FOR PLATE PACK

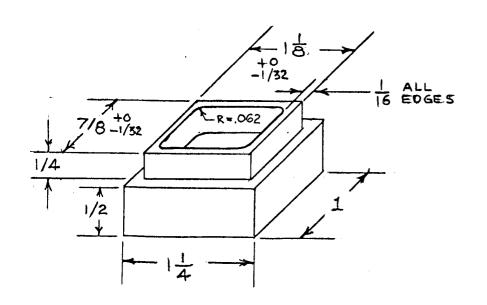
BY FSC DATE 4-6-65 CHKD. BY DATE

SUBJECT PLASTIC BELLOWS
FOR SEALED SILVER CADMIUM
CELL OF 8 A.H. RATED CAP

SHEET NO. 1 OF 1
JOB NO. 83276
SK-83276-T2-10

FIGURE 24





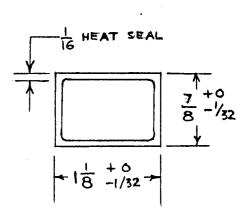
B - HEAT SEALING DIE FOR PLASTIC PILLOW

MATERIAL = BRASS

USE 3/4 × 1

RECTANGULAR

STOCK NO. 31-167



C - PLASTIC PILLOW FOR

MULTI-SECTION RECT
ANGULAR BELLOWS

MATERIAL = POLYPROPYLENE
FILM - 4 MILS
THICK PROD. BY
AVISUN CO.

NEG.

1L EM-476 RETAINER

1L EM-476 FROM POS.

IN SIMPLE U-FOLD

BY FSC	DATE
CHKD, BY	DATE

CONCEPTUAL

SUBJECT BELLOWS - SWITCH ASS'Y

AND INSTALLATION IN BAH SEALED

SILVER CADMIUM CELL.

SHEET NO. 1 OF 1	
JOB NO. 83276	
SK-T3-1	

FIGURE 26

